

# PATENT SPECIFICATION

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## (54) METHOD OF PHOSPHATING METAL SURFACES

(71) We, VAQ-FOS LIMITED, a British Company, of High Street, Henley-in-Arden, in the County of Warwickshire, do hereby declare the invention, for which we pray that a Patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention is concerned with a method of phosphating metal surfaces, i.e. treating the surfaces of metal objects with aqueous acidic coating solutions containing phosphates to form phosphate coatings thereon.

Phosphating is performed for a number of purposes. One purpose is the provision on metal surfaces of a lubricant or a carrier for lubricant, for example soap, and this has the following applications:—

1. in the preparation of metal billets, slugs or blanks for cold extrusion processes;
2. in the preparation of die blanks for cold hobbing processes;
3. in the preparation of wire, rod, bar or tube for cold drawing processes; and
4. in the surface treatment of gear wheels, camshafts, cylinder liners, transmissions components, connecting rods and other moving parts having sliding surfaces to provide or assist lubrication thereof particularly when they are being run in.

Another purpose of phosphating is the preparation of metal surfaces for painting or plastic coating of the surfaces and this has the following applications:—

1. in the pre-treatment of vehicle bodies, bicycle and motor-cycle frames, refrigerator cabinets and washing machine and similar cabinets to improve paint adhesion and corrosion protection, and
2. in the pre-treatment of metal sheet and strip to improve the adherence of paint or plastic coatings subsequently applied thereto.

Yet another purpose is the provision of a corrosion-resisting layer on a metal surface.

Conventional methods of phosphating metal surfaces have several disadvantages. They are time-consuming and hence expensive. A large

number of different phosphating solutions of complex compositions have to be provided for different applications. The phosphate coating formed on a surface is likely to be unsatisfactory, for example the size of the phosphate crystals and the crystalline structure may be such that the coating has an unacceptable degree of porosity and lacks sufficient corrosion resistance and the bond between the coating and the metal surface may be weak.

In order to illustrate the disadvantages of conventional methods of phosphating metal objects, detailed reference will be made to one such method, namely the preparation of a metal billet for cold extrusion thereof, which includes the steps of barrelling the billet to descale and deburr it, pickling the billet in two successive acid baths to clean and degrease its surface in preparation for phosphating, subjecting the billet to successive cold and hot water rinses usually by dipping it in hot and cold water baths to remove the acid from its surface, soaking the billet in a phosphating solution for a period sufficient to provide a layer of phosphate of the required coating weight on the surface of the billet, and finally subjecting the billet to successive cold and hot water rinses usually by dipping.

This particular method has a number of disadvantages. It is difficult accurately to control the strength of the pickling acid, the condition of the pickled surface being a major factor affecting the quality of the final phosphate coating, and problems also arise in disposing of the acid effluent.

During transfer of the billet from the pre-treatment steps to the phosphating step its surface may deteriorate and the full effect of the pretreatment may be lost with the result that the quality of the phosphate coating is affected. The method involves several separate steps and is lengthy and therefore expensive, the periods of the respective steps being as follows depending on the initial condition of the surface to be treated:—

	Barrelling	10—15 minutes
	Acid Degreasing	10—15 minutes
	Acid Pickling	5—20 minutes
	Cold Water rinse	2 minutes
5	Hot water rinse	2 minutes
	Phosphating	10—12 minutes
	Cold water rinse	2 minutes
	Hot water rinse	2 minutes

10 It is an object of the present invention to provide an improved method of phosphating metal surfaces.

15 According to the invention we provide a method of treating a metal surface to provide a phosphate coating thereon, comprising subjecting the surface to a blast of a mixture of abrasive particles and an aqueous acidic solution containing a phosphate, the mean velocity of impact (as hereinafter defined) of the particles of the blast with the surface being between 20 and 100 feet per second.

20 By "mean velocity of impact" we mean the middle or mean value between the velocity of the slowest particle in the central area of the blast as the particle impacts the surface and the velocity of the fastest particle in said central area as the particle impacts the surface. By the central area of the blast we mean that part of the blast which impacts the surface within an area which is symmetrical about the centre of the blast pattern, said area being half the total area of the blast pattern.

25 There are several important properties required by a phosphate coating and the balance of these properties will depend on the purpose for which the phosphate coating has been applied to a metal surface, such purposes being exemplified above. The mean impact velocity has been found to be the controlling factor to give a good balance of all the required properties.

30 One of the properties is the coating weight, i.e. the weight of the phosphate coating which is normally measured in milligrams per square foot. The coating weight for different applications varies. Thus, for example, a zinc phosphate coating applied to a billet for the purpose of preparing it for cold extrusion may be required to have a coating weight of 700 milligrams per square foot whereas a coating weight of 350 to 400 milligrams per square foot may be sufficient for preparing a metal sheet for a light drawing operation. For preparing a metal surface for painting it is preferred that the coating weight be below 600 milligrams per square foot and on average between 250 and 300 milligrams per square foot.

35 Whatever the coating weight required, however, it is desirable that this coating weight be achieved in as short a time as possible. Moreover, we have found that for a given time during which the surface is exposed to the blast, a given concentration of the phosphating solution and a given temperature of the solution, the coating rate will depend on the impact velocity of the blast. If the velocity is too low then it will take longer than the optimum time to produce a coating of the desired weight whereas if the velocity is too high we have found that the coating is removed by the abrasive particles more rapidly than it is formed, so that there is a falling off in the coating weight.

65 Another important property of the coating is its resistance to corrosion and here again we have found that, if a surface is exposed to the blast for a given period and other conditions are equal, the corrosion resistance is not at its optimum value if the impact velocity of the blast is too low but that it also decreases from its optimum value if the impact velocity of the blast is too high.

70 A third important feature of the coating is its porosity and again we have found again that, if a surface is exposed to the blast for a given time and other conditions are equal, there is an optimum range of impact velocities within which the coating is least porous.

75 Taking all these properties into account, we have found as a result of experiment that the impact velocity of the blast should lie within the range set forth above, i.e. between 20 and 100 feet per second.

80 The mean impact velocity as defined above can be determined by photographic techniques. Thus, photographs of the blast can be taken with a very high speed camera under appropriate lighting conditions so that individual particles or groups of particles can be identified in the photographs. We have found that by photographing the blast at film speeds of 5000 frames per second when the blast is illuminated from behind by a 7 kilowatt flood-lamp, good results can be obtained. When the film is projected on to a screen, one can measure the distance on the screen which a particle has moved in the 1/5000 second which has elapsed between two adjacent frames. One can then apply a correction figure for the magnification and thereby arrive at the actual distance moved by the particles. In measuring the particle velocity it is preferred that the blast passes freely past the point of measurement.

85 The phosphating solution in the blast is preferably atomised by means of compressed air and the air pressure can be varied to vary the mean impact velocity of the particles.

90 If the surface is to be descaled as well as given a phosphate coating then it is preferred that the mean impact velocity as defined above shall be between 40 and 100 feet per second, preferably between 50 and 80 feet per second.

95 Any scale and grease present on the surface is removed by the abrasive which produces a nascent surface with which the phosphating solution reacts chemically without impediment,

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thereby producing a phosphate layer on the surface. Spent abrasive particles and detritus are carried away from the surface by the solution as it flows away from the surface.

5 The final coating weight of phosphate per unit area is dependent on the concentration and temperature of the phosphating solution, the time during which the process is performed, and the mean impact velocity of the blast.

10 The effects of concentration and temperature of the phosphating solution and time are substantially what would be expected from conventional phosphating methods. Thus, the greater the treatment time the heavier the coating weight up to a limiting value, assuming other conditions of the process are constant. The greater the concentration of the phosphating solution again the greater the coating weight, assuming other conditions are equal. The higher the temperature of the phosphating solution then again, other conditions being equal, the greater the coating weight. The feature which could not have been predicated is the effect of the mean velocity of impact of the blast on the surface and the discovery that, if the velocity is too high the abrasive takes off more of the phosphate coating than is formed by the phosphate solution.

30 The phosphating solution used in the method will be an acidic solution of phosphates in water in proportions depending on the degree of dilution of the phosphate required. The solution may include sodium nitrite or other agents which act as accelerators and/or oxidising agents for the reaction of the phosphating solution with the surface.

40 An advantage of the invention is that only one phosphating solution is required for the wide range of applications of phosphating, as compared with the large number needed in conventional methods. In fact, we have found that the best results in a large number of applications are obtained with the use of the phosphating solution conventionally used for preparing a metal billet for cold extrusion. Such a solution has a sodium nitrite accelerator and is not normally recommended for applications such as pre-painting preparation where a fine coating is required, since it generates sludge and with sludge present a coarse non-uniform coating is produced. However, this problem does not arise with the present invention since the continuous flow of phosphating solution over the surface prevents a build-up of sludge.

55 Preferably the method is performed using a hot solution which will accelerate the reaction of the phosphate with the metal surface.

60 The process will be advantageously performed in as short a time as possible since the sooner the phosphate coating is applied to the surface after the latter had been made nascent and the quicker the phosphate crystals

grow to form the coating, the better the quality and adherence of the coating.

It is an advantage of the invention that the treatment can be performed more quickly than in conventional phosphating methods. For example, a metal billet can be descaled, cleaned, degreased and phosphated in one operation by the method of the invention in preparation for cold extrusion thereof in as little as one minute compared with 33—35 minutes taken by the conventional method described above.

A metal billet should not require any treatment before being subjected to the method according to the invention since the method itself is normally sufficient to prepare the surface of the billet for phosphating. However, where the billet has been sawn from a parent bar it may have to be barrelling or otherwise treated so that any excessive burrs produced by the cutting, which would not be removed by the abrasive particles, are removed.

The properties of resistance to corrosion and porosity of a phosphated surface depend on the size of the phosphate crystals and the crystalline structure and a further advantage of the method according to the invention as compared with conventional phosphating methods is that it produces smaller crystals and a finer crystalline structure with the result that the phosphate coating provides a more effective protection against corrosion and has less porosity than coatings produced by the conventional methods. The reason for this is that in conventional methods the crystals have comparatively few reaction sites on the surface and therefore grow to a relatively large size as they spread out from the sites over the surface, whereas in the method of the invention the abrasive particles in the blast form a considerably larger number of reaction sites for the crystals at the locations where they impinge upon the surface and as a result the crystals grow close together in a fine structure and are hence comparatively small. It is thought that a fine structure of small crystals also improves the lubricity of the phosphate coating as compared with coatings produced by conventional phosphating methods.

Further advantages of the method according to the invention, as compared with conventional methods, are that a firmer bond is produced between the phosphate coating and the metal surface since there is no surface-contaminating layer such as an oxide or hydroxide and, since the phosphate crystals are smaller, the coating weight can be lower in certain cases to obtain the same effect.

If a heavier phosphate coating is required than can be obtained solely with a blast containing abrasive particles and a phosphating solution the method is continued using a phosphating solution without abrasive to increase the weight of the coating. For example, phosphating solution may be sprayed on to

the metal object or the object may be dipped in a bath of phosphating solution.

If desired, the surface after phosphating thereof may be subjected to a clean water rinse.

The mixture of the phosphating solution and abrasive particles is conveniently collected after use, at least partially cleaned and then reused. Thus, the mixture may be continuously circulated and reused in the treatment of a succession of metal objects.

In a specific embodiment of the invention applied to the treatment of a succession of metal objects, on each circuit of the mixture all the abrasive particles and detritus are separated therefrom and then a part of the cleaned solution is removed from the flow and passed, by a separate route, to a metal object which has been blasted with a mixture of abrasive particles and a phosphating solution thereby to rinse the object, the remainder of the cleaned solution being remixed with abrasive particles and then directed against a subsequent object.

The method of the invention is preferably performed in an apparatus comprising a chamber which contains means, for example a nozzle, for directing the blast against the surface of a metal object and the lower part of which is formed as a sump for collecting slurry, and means for continuously feeding slurry from the sump to the blast-directing means. Propulsion of the blast against the surface is preferably effected by compressed air supplied to the blast directing means.

The chamber may be capable of being closed and provided with means enabling it to be opened to facilitate the introduction therein and the removal therefrom of an object or batch of objects, or means may be provided for continuously feeding objects through the chamber. In any event the chamber will be made of, or lined with, material such as stainless steel or polyvinylchloride which will not become heavily phosphated and which will resist attack by the acidic phosphate solution.

The chamber preferably contains means, for example, spray heads for directing phosphating liquid against each object after wet blasting thereof.

In a specific embodiment of the apparatus the sump is connected through a pump to a centrifugal separator which has connections to a filter unit, a nozzle and a first rinsing spray head. The arrangement is that the separator separates coarse abrasive particles and detritus from the used slurry fed to the separator from the sump by the pump and a part of the slurry is then drawn off and fed to the filter unit where fine particles and detritus are separated from the liquid. The filter unit includes means for heating the liquid and is connected through a pump to a second spray head in the chamber. A partially

cleaned mixture of a phosphating liquid and abrasive particles is fed from the separator to the first spray head and cleaned phosphating liquid is fed from the filter unit to the second spray head, so that each object is subjected to two rinses.

The chamber may also contain a spray that connected to a water supply for applying a final rinse to each object.

Reference will now be made to the accompanying graphs which show examples of results achieved by the use of the method of the invention, and to the accompanying photographs which show examples of phosphate crystalline structures produced by the said method and by a conventional phosphating method.

The graphs were obtained using blasts in which the abrasive particles were alumina, of angular shape and of 90/100 grit size. The concentration of the abrasive-phosphating solution mixture in each case was 40 lb. of abrasive in 25 gallons of the appropriate solution.

The curves which will be described were obtained by averaging test results in which a metal surface was blasted with an abrasive-phosphating solution mixture for 30 seconds followed by a 30 second rinse with a phosphating solution without abrasive particles therein.

The velocity of the blast was measured by the photographic method described above and the velocity was varied by changing the pressure of the compressed air used to atomise the liquid and to propel the blast at the surface. The amount of slurry which was pumped by the pump to the spray nozzles was kept constant at 25 gallons per minute.

Although the specific examples described below use zinc phosphate, iron phosphate, manganese phosphate and lead phosphate alone, the invention may be carried out using any desired combination of these phosphates in a manner which will be clear to a person skilled in the art.

#### Example 1

##### Zinc Phosphate

The solution used was a proprietary zinc phosphate of the following specification:—

Phosphoric Acid Radical	27 gm/litre	115
Zinc	2.1 gm/litre	
Sodium nitrite		
accelerator	0.15 gm/litre	
pH	2.0—202	
Total acid content		120
(pointage)	70	
Free acid content	4	
Operating temperature	70°C	

Referring now to Graph 1 this shows the effect on the coating weight of the mean velocity of impact of the blast with the metal surface. The coating weight analysis was made

by stripping the coating and chemically determining the phosphate contact rather than by the less accurate weight difference technique. the curve marked 10 is that obtained on the surface of a scaled plate and the curve marked 11 is that obtained on the surface of a bright rolled plate. It will be seen that in order to obtain good coating weights within the time of the test it was necessary for the mean impact velocity to be between 20 and 100 feet per second with an optimum range of between 60 and 100 feet per second. Any increase in velocity over 100 feet per second reduces the coating weight since the abrasive removes more phosphate coating than is formed by the phosphating solution.

Graph 2 shows the effect of the mean impact velocity on corrosion resistance. Corrosion resistance is measured in hours and the test is a neutral salt spray test which is carried out at 35°C. in accordance with the ASTM B117 specification in which the phosphate coating is sealed with a standard lanolin based mineral oil. The onset of rusting is determined by an experienced operator.

It will be seen that the corrosion resistance is at a maximum at a mean impact velocity of approximately 60 feet per second but that good corrosion resistance is obtained within the velocity range of 20 to 100 feet per second. Impact velocities greater than 100 feet per second cause the corrosion resistance to decrease.

Referring now to Graph 3, this contains two curves 12 and 13, the curve 12 being obtained by treating bright rolled plate and

the curve 13 being obtained by treating scaled plate. The porosity is measured using a ferroxyl test which is carried out as follows.

Ferroxyl reagent is prepared freshly by dissolving 30 g. of sodium chloride and 1 g. of potassium ferrocyanide in water and making up the resulting solution to 1 litre. The test piece is degreased by immersion for at least one minute in trichloroethylene at room temperature. A filter paper, 9 cm. in diameter, is immersed for ten seconds in the ferroxyl reagent and is allowed to drain for 20 seconds. The filter paper is then placed so that it lies flat on a horizontal surface of the test piece. After one minute the filter paper is removed and is dried by placing between two further filter papers. Where the ferroxyl reagent has been in contact with the bare metal the filter paper will have changed colour in small areas which correspond to the pores in the surface coating. The total area of the pores is compared with previously prepared standards corresponding to five arbitrary grades of porosity from 0 which indicates no porosity, to 4 which is the most porous.

The ratings are made by the operator and it will be seen that the least porosity is obtained at impact velocities around 60 feet per second but that satisfactory values are obtained by means impact velocities of between 20 and 100 feet per second.

#### Example 2

##### Iron Phosphate

The solution used was a proprietary iron phosphate of the following specification:—

Phosphoric Acid Radical	19.4 gm/litre
Iron Radical	2.9 gm/litre
Pointage	30
Free acid content	3—5
Operating Temperature	75°C (higher temperatures up to 99°C will give a faster build-up rate).

Graph 4 shows the effect of the mean impact velocity on the coating weight obtained with bright drawn plate. It will be seen that in order to obtain good coating weights within the time of the test it is necessary that the mean impact velocity be between 20 and 100 feet per second and for optimum results between 40 and 100 feet per second. Any increase in velocity over 100 feet per second reduces the coating weight since the abrasive removes more phosphate coating than is formed by the solution.

#### Example 3

##### Manganese Phosphate

The solution used was a proprietary

manganese phosphate of the following specification:—

Phosphoric Acid Radical	25.9 gm/litre
Manganese	5.2 gm/litre
Iron II	2.3 gm/litre
Nitrite radical	9.4 gm/litre
Pointage	20
Free Acid content	3.3—3.6
Operating temperature	95°C

Graph 5 shows the effect of the mean impact velocity of the blast on the coating weight obtained with bright drawn plate. It will be seen that in order to obtain good coating weights within the time of the test

it is necessary for the mean impact velocity to be between 20 and 100 feet per second with an optimum range of between 40 and 100 feet per second. Any increase in velocity over 100 feet per second reduces the coating weight since the abrasive removes more coating than is formed by the solution.

#### Example 4

##### Lead Phosphate

- 10 The solution used was a proprietary lead phosphate solution of the following specification:—

	Pointage (free acid content)	30—40
15	Lead Pointage	8—10
	Operating temperature	30°C

- Graph 6 shows the effect of the mean impact velocity on the coating weight obtained with bright drawn plate. It will be seen that in order to obtain good coating weights within the time of the test it is necessary for the mean impact velocity to be between 20 and 100 feet per second with an optimum range of between 40 and 100 feet per second. Any increase in velocity over 100 feet per second reduces the coating weight since the abrasive removes more coating than is being formed by the solution.

- As examples of the applications of these phosphating solutions there are the preparation of metal billets for cold extrusion processes in the case of zinc, the preparation of metal surfaces for painting in the case of both iron and lead, the surface treatment of gears, splines and other moving parts having sliding surfaces to provide a lubricant on said surfaces in the case of both manganese and lead, and the provision of corrosion-resistant layers on metal surfaces in the case of lead.

- 40 Analysis of the phosphating solutions involves testing for the total acid content (pointage) and/or, the free acid content of each solution and in the case of manganese phosphate the iron II content as well. The tests involve standard titration techniques using different indicators and reagents depending on the solutions involved.

- The pointage is required to be determined in the case of iron, manganese and zinc only. In a specific example applied to a zinc phosphate solution a 10 ml. sample of solution is titrated against N/10 sodium hydroxide solution using about 5 drops of phenolphthalein indicator. A colour change from colourless to pink determines the end-point of the titration. A working solution requiring for example 70 ml. of N/10 sodium hydroxide to reach the end-point is known as a 70 point solution.

- 60 The free acid content is required to be determined for manganese and lead only. In the case of a manganese phosphate solution

10 ml of solution is added to 10 ml of distilled water and titrated against N/5 sodium hydroxide with about 5 drops of bromophenol blue indicator. A colour change from yellow to bluish green determines the end-point. The amount in ml. of N/5 sodium hydroxide required to reach the end-point is the degree of free acid.

In the case of lead phosphate solution a 2 ml sample is titrated against N/10 sodium hydroxide with 3—4 drops of modified methyl orange indicator. A colour change from pink to green determines the end-point. The number of mls. multiplied by 5 of N/10 sodium hydroxide required to reach the end-point is equal to the free acid level.

To determine the iron II content of manganese phosphate solution a 10 ml sample with 10 to 20 drops of phosphoric/sulphuric acid solution is titrated against N/10 potassium permanganate. A colour change from colourless to pink which persists for about 30 seconds determines the end-point of the titration. The percentage of iron II is equal to the number of mls. of N/10 potassium permanganate required to reach end end-point divided by 18.

Referring now to the accompanying phosphates, Figure 1 shows at a magnification of 550 the crystalline structure of a phosphate coating produced by a method according to the invention and Figure 2 shows at the same magnification the crystalline structure of a coating produced by a conventional phosphating method using the same chemical. It will be observed from a comparison of these photographs that the coating produced by the invention has smaller crystals and a finer crystalline structure than the coating produced by the conventional method.

#### WHAT WE CLAIM IS:—

1. A method of treating a metal surface to provide a phosphate coating thereon, comprising subjecting the surface to a blast of a mixture of abrasive particles and an aqueous acidic solution containing a phosphate, the mean velocity of impact (as herein defined) of the particles of the blast with the surface being between 20 and 100 feet per second.

2. A method as claimed in Claim 1 wherein the mean velocity of impact of the particles of the blast with the surface is between 40 and 100 feet per second whereby the surface can be descaled as well as phosphated.

3. A method as claimed in Claim 2 wherein the mean velocity of impact of the particles of the blast with the surface is between 50 and 80 feet per second.

4. A method as claimed in any one of the preceding claims wherein the solution in the blast is atomised by means of compressed air.

5. A method as claimed in Claim 4 wherein the pressure of the compressed air is varied

to vary the mean velocity of impact of the particles with the surface.

5 6. A method as claimed in any one of the preceding claims wherein the solution includes an accelerator and/or an oxidising agent for the reaction of the solution with the surface.

10 7. A method as claimed in Claim 6 wherein the solution contains a sodium nitrite accelerator.

8. A method as claimed in any one of the preceding claims wherein the solution is heated.

15 9. A method as claimed in any one of the preceding claims which includes treating the surface with a phosphating solution without abrasive particles after subjecting the surface to the blast.

20 10. A method as claimed in Claim 9 wherein the phosphating solution without abrasive particles is sprayed on to the surface.

25 11. A method as claimed in any one of the preceding claims which includes subjecting the surface to a clean water rinse after phosphating thereof.

30 12. A method as claimed in any one of the preceding claims wherein the mixture of the phosphating solution and abrasive particles is collected after use, at least partially cleaned and then reused.

13. A method as claimed in Claim 12 wherein the mixture is continuously circulated and reused in the treatment of a succession of metal objects.

35 14. A method as claimed in Claim 13 wherein on each circuit of the mixture all the abrasive particles and detritus are separated therefrom and then a part of the cleaned solution is removed from the flow and passed, by a separate route, to a metal object which has been blasted with a mixture of abrasive particles and a phosphating solution thereby to rinse the object, the remainder of the cleaned solution being re-mixed with abrasive particles and then directed against a subsequent object.

40 15. Methods according to Claim 1 of treating metal surfaces to provide phosphate coatings thereon, substantially as hereinbefore described.

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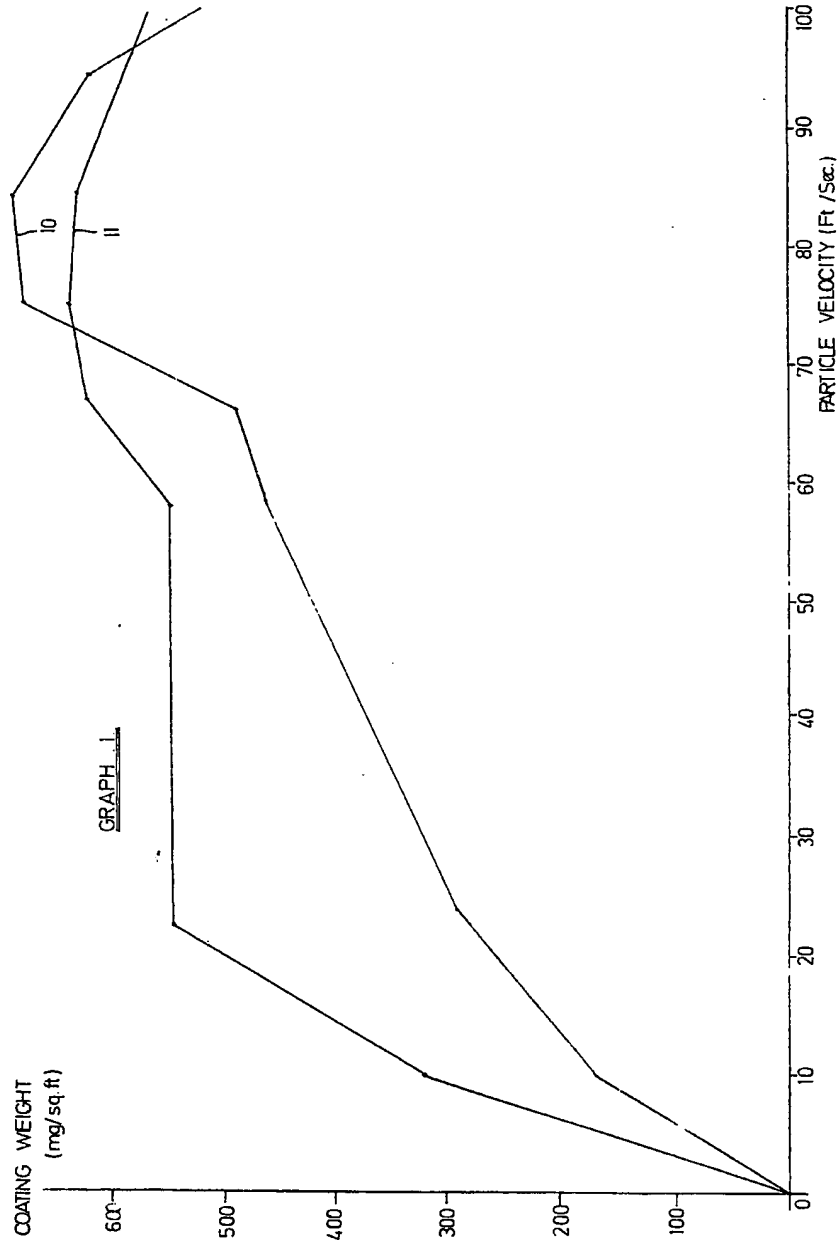
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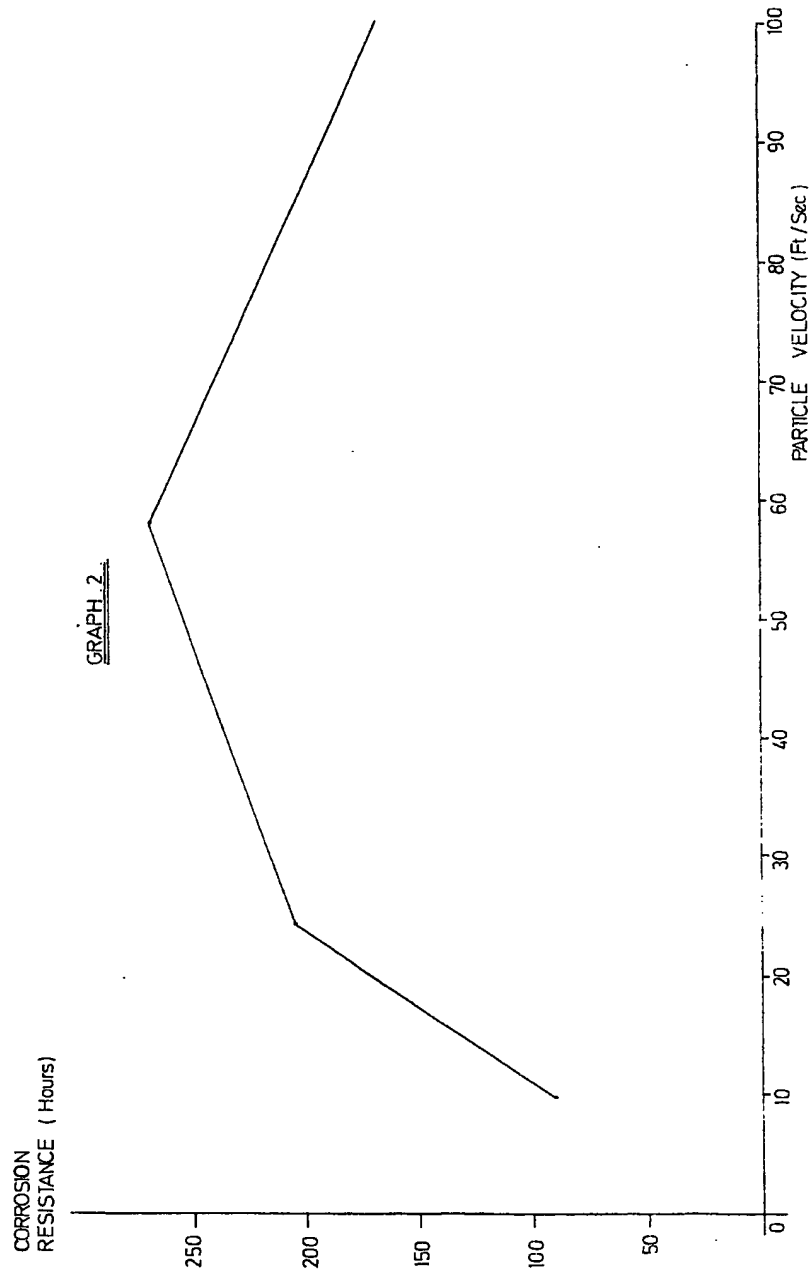
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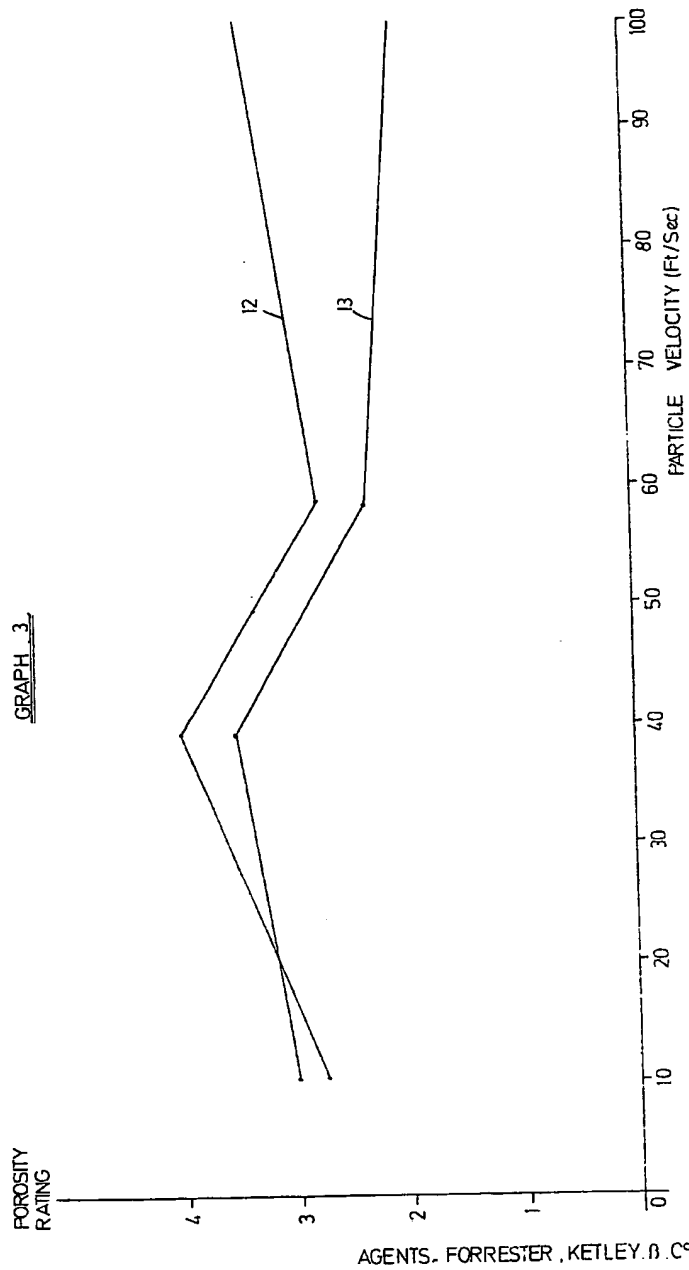
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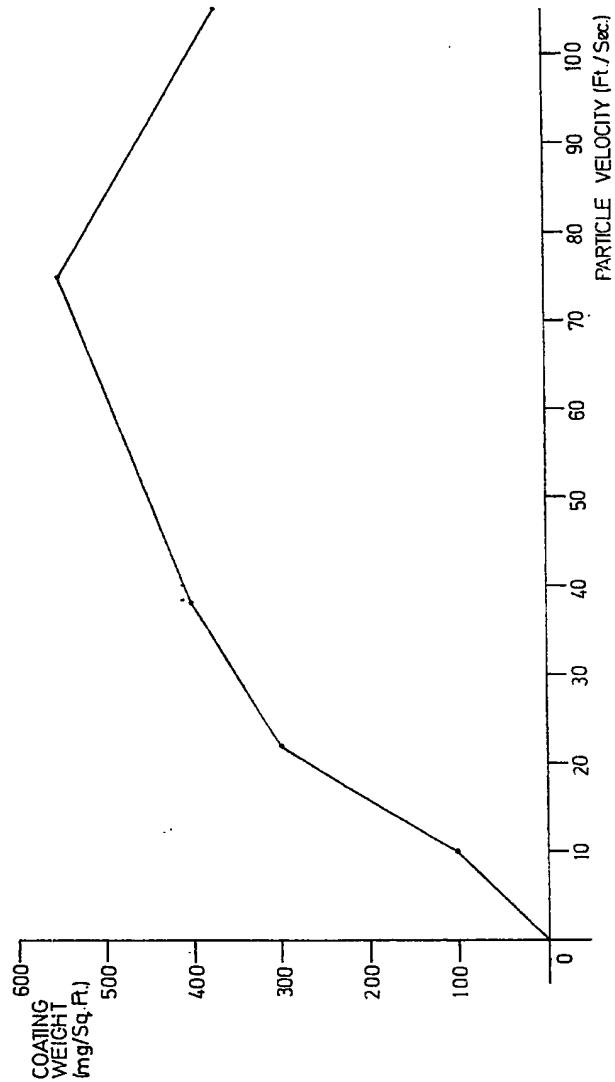
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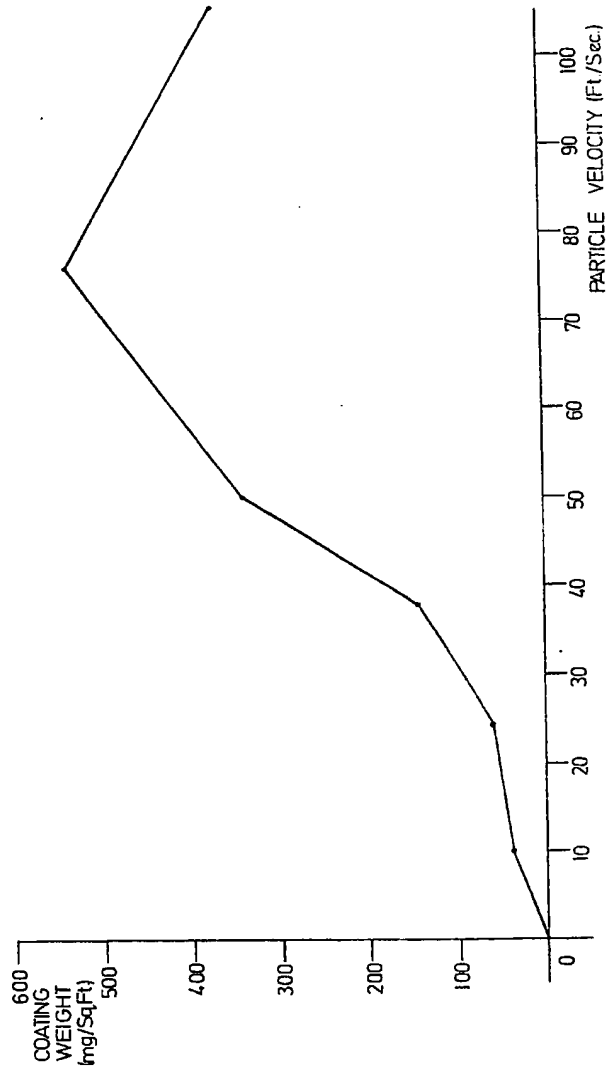
Sheet 3



GRAPH 4



GRAPH 5



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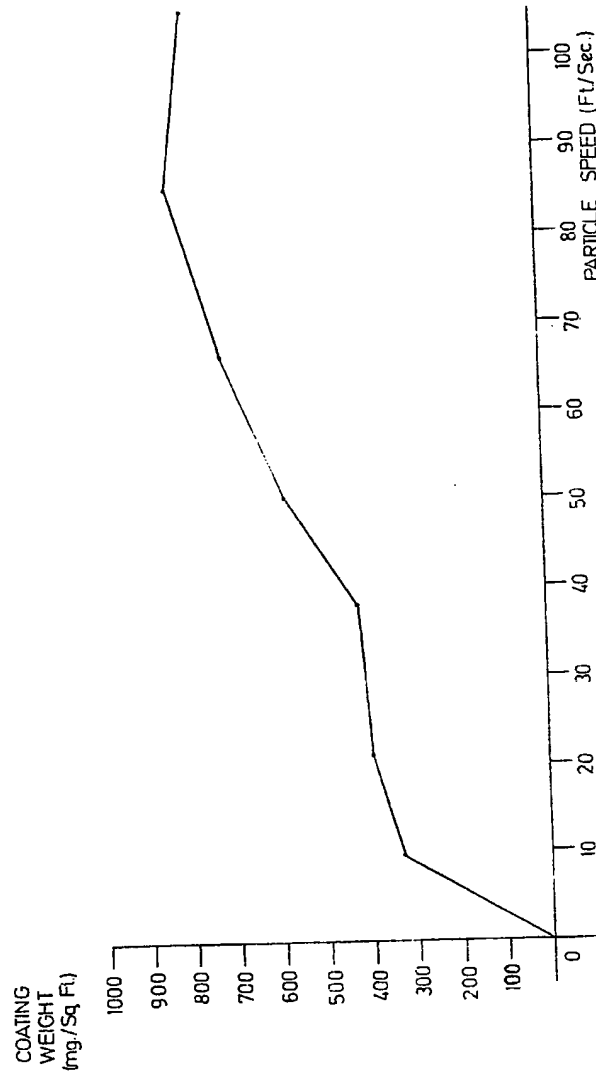
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Sheet 6

GRAPH 6



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FIG. 2.



FIG. 1.

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